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## The molecular configuration of 1,4-dimethyl-5-iminotetrazole.

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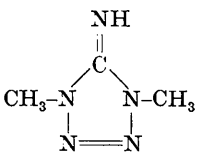
The unexpected results obtained from the crystal structure analysis of the hydrochloride and hydrobromide salts of 1,3-dimethyl-5-iminotetrazole (Bryden, Henry, Finnegan, Boschan, McEwan & Van Dolah, 1953; Bryden, 1955) made it desirable to check the configuration of the isomeric 1,4-dimethyl-5-iminotetrazole,



Accordingly, crystals of the hydrochloride and hydrobromide salts of this compound (Henry, Finnegan & Lieber, 1954) were examined, and a partial structure analysis of the hydrobromide salt was made. As no further work on these compounds is planned, the results obtained are reported here.

Crystals of the hydrochloride salt of 1,4-dimethyl-5-iminotetrazole are orthorhombic. The dimensions of the unit cell are

$a_0 = 16{\cdot}32$, $b_0 = 8{\cdot}10$, $c_0 = 5{\cdot}54$ Å ($\lambda$ of Cu $K\alpha = 1{\cdot}5418$ Å),

giving a calculated density of 1·356 g.cm.$^{-3}$ with 4 molecules per unit cell. The space group is $P2_12_12_1$.

Crystals of the hydrobromide salt were found to be monoclinic, having the unit-cell dimensions

$a_0 = 19{\cdot}82$, $b_0 = 5{\cdot}08$, $c_0 = 16{\cdot}90$ Å, $\beta = 117° 40'$
($\lambda$ of Cu $K\alpha = 1{\cdot}5418$ Å).

The density calculated for 8 molecules per unit cell is 1·710 g.cm.$^{-3}$. The space group is either $Cc$ or $C2/c$ (the extinctions observed are $hkl$ present only with $h+k = 2n$ and $h0l$ present only with $l = 2n$), the latter probably being correct.

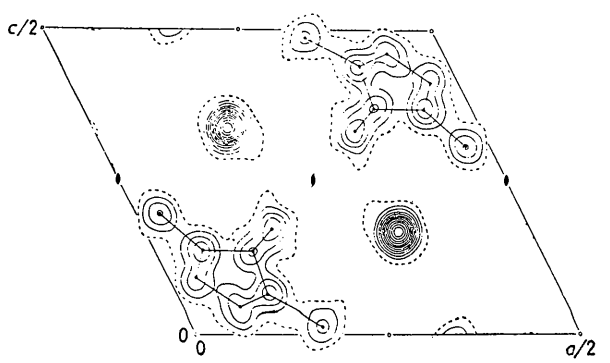



Fig. 1. Second Fourier projection on (010) of the hydrobromide salt of 1,4-dimethyl-5-iminotetrazole.

The intensities of the $h0l$ reflections from a crystal of the hydrobromide salt were estimated in order to obtain the configuration of the molecule. This approach seemed more expedient than using the hydrochloride salt since the heavy bromide ion will determine the phases of the majority of the reflections. The bromide ion was easily located from the Patterson projection on (010). The contribution of this ion to all the $h0l$ reflections was then used to determine the phases, and a Fourier projection was calculated with all terms having reasonably certain signs. This first Fourier projection clearly resolved all atoms in the molecule. The second Fourier projection, calculated with 157 out of a possible 192 $h0l$ terms (only 159 were observed), is shown in Fig. 1. It is immediately seen that the molecule has the expected 1,4-configuration. Hydrogen bonds between the imino groups and the halogen ions form infinite spirals along the twofold screw axes. A similar arrangement probably exists in crystals of the hydrochloride salt, as twofold screw axes are the only symmetry elements present. This differs from the crystal structure of the 1,3-compound where the hydrogen bonding occurs in the mirror planes containing the molecules. The spiral arrangement along the $b$ axis is consistent with the observation that crushing the crystals causes them to cleave along the $b$ axis into bundles of fibers. Because of the tilted arrangement of the molecule with respect to the plane of projection, no attempt was made to estimate bond lengths. For the last set of $h0l$ structure amplitudes calculated, the value of the disagreement quotient, $R$, was 0·23. In these calculations an isotropic temperature correction,

$$\exp\left[-5{\cdot}025\,(\sin\theta/\lambda)^2\right],$$

was used.

The compounds used in this investigation were obtained from Drs W. G. Finnegan and R. A. Henry of this Laboratory.

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### References

BRYDEN, J. H. (1955). *Acta Cryst.* **8**, 211.

BRYDEN, J. H., HENRY, R. A., FINNEGAN, W. G., BOSCHAN, R. H., MCEWAN, W. S. & VAN DOLAH, R. W. (1953). *J. Amer. Chem. Soc.* **75**, 4863.

HENRY, R. A., FINNEGAN, W. G. & LIEBER, E. (1954). *J. Amer. Chem. Soc.* **76**, 2894.